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Attorney Docket No.: TS7564 (US) A Named Inventor/Application Identifier: Cameron W. Watson Express Mail Label No.: <u>FL 28/13729345</u>
Title: HYDRAULIC FLUID

Date: August 25, 2000



UTILITY PATENT APPLICATION TRANSMITTAL UNDER 37 CFR 1.53(b)

Assistant Commissioner for Patents Box Patent Applications Washington, DC 20231

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(37 CFR 1.16(c))				
INDEPENDENT CLAIMS	2 - 3 =	0	X \$78.00 =	0
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Respectfully submitted,

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TS 7564 (US)

HYDRAULIC FLUID

The present invention relates to a hydraulic fluid.

Background of the Invention

WO 93/03121 relates to hydraulic fluids having improved wet filterability and having increased resistance to loss of zinc when the oil is exposed to water. These compositions comprise (A) a combination of a metal salt selected from the group consisting of sulfonates, phenates, carboxylates and mixtures thereof, (B) an aliphatic carboxylic acid or anhydride, or carboxylic acid group containing derivative thereof, wherein the aliphatic group contains at least 20 carbon atoms, and optionally (C) a metal salt of (C)(I) at least one organic phosphorus acid or mixture of (C)(I) at least one organic phosphorus acid and (C)(II) at least one carboxylic acid group, and optionally (D) triazole. In one embodiment, the carboxylic acids of (A) are aromatic carboxylic acids. Zinc, calcium and magnesium salts of these aromatic carboxylic acids, and especially salicylic acids, are preferred. Although magnesium salicylate and zinc dithiophosphate are both mentioned per se, there is no teaching to use the combination of these compounds.

WO 89/04358 relates to compositions comprising (A) a neutral or basic metal salt of an acidic organic compound, (B) a metal deactivator, preferably an optionally substituted benzotriazole, and (C) a sulfur and/or phosphorus-containing compound selected from the group consisting of (C-1) phosphorus-containing amide, (C-2) phosphorus-containing ester, (C-3) sulfur-coupled

dithiocarbamate, (C-4) sulfur-containing compound according to formula (I). Particularly preferred metals for the salts (A) are sodium, magnesium, calcium or mixtures of two or more thereof. In the extensive list of additional compounds which can be present, zinc dialkyl dithiophosphate is mentioned. Although magnesium salicylate and zinc dithiophosphate are both mentioned per se, there is no teaching to use the combination of these compounds.

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EP-A-604 218 describes functional fluids comprising a major amount of an oil of lubricating viscosity, at least one metal salt of thiophosphoric acid, a zinc salt of salicylic acid, and a rust inhibitor, which fluid contains less than about 1 %wt of dispersant and less than about 1 %wt of basic sulfonate salt. The thiophosphoric acid is most commonly a dihydrocarbyl dithiophosphoric acid; the metal is most preferably zinc. The teaching of the document is restricted to the use of zinc salicylate.

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In US-A-4,627,928, a process has been described for preparing basic magnesium salicylates which can be characterised as having a magnesium content of at least 150% up to 500%, of the stoichiometrically equivalent amount of magnesium based on the amount of total acid present. The magnesium salts obtained are described to be suitable for use as additives in general, more specifically as additives for a variety of lubricating oils and fuels, e.g. gasoline and diesel fuels. It is mentioned that these specific magnesium salicylates can be used in combination with a wide range of other compounds. There is no disclosure or teaching to apply a

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combination of zinc dithiophosphate and magnesium salicylate in a hydraulic fluid.

Hydraulic fluids need to meet special requirements as they both lubricate and transmit pressure. In order to meet these requirements, hydraulic fluids contain additives. Generally, hydraulic fluids contain antiwear/extreme pressure additives. The conditions for a hydraulic fluid are especially difficult in vane pumps where the vane tips slide against the casing at high speed under heavy load and at high temperature. Further, the increasingly high operating temperatures of modern hydraulic equipment requires the fluid to be thermally stable to avoid the formation of deposits and sludge and to resist the corrosion of both ferrous and non-ferrous metals. The requirement of good thermal stability in hydraulic fluids containing an anti-wear additive, means that these additives are often used in combination with detergents. However, the calcium salts of alkylbenzene sulphonates, alkyl-naphthalene sulphonates, petroleum sulphonates, alkylphenates, alkyl sulphurized phenates or alkylsalicylates which are usually applied, give increased wear. The increased wear is especially marked in vane pumps, more specifically under low load conditions. Low load conditions occur in practice when the pump is idle.

Summary of the Invention

It has now surprisingly been found that hydraulic fluid containing a combination of magnesium salicylate and zinc dithiophosphate, gives improved performance at low load compared with a combination containing calcium salicylate. Additionally, it was found that this combination also provides greater thermal stability. Less

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sludge and deposits are formed when subjecting the hydraulic fluid of the present invention to the Cincinnati Milacron Thermal Stability Test.

The present invention provides a hydraulic fluid comprising a lubricant base oil in combination with (a) from 0.001 to 5 %wt of magnesium salicylate, (b) from 0.01 to 8 %wt of zinc dithiophosphate, which amounts are based on total weight of hydraulic fluid.

Detailed Description of the Invention

The magnesium salicylate for use in the present invention can be either neutral or overbased. expression "overbased" is equivalent to "basic", "superbased", "hyperbased" and "high-metal containing salts". These magnesium salicylates contain an excess metal content compared to the amount of metal which would be present according to the stoichiometry of the metal and the salicylic acid reacted with the metal. Processes for making such neutral and basic metal salts are well known in the art. Neutral salts can be made by heating a mineral oil solution of an acidic organic compound with a stoichiometric equivalent amount of a metal neutralizing agent such as the metal oxide, hydroxide, carbonate, bicarbonate, or sulfide at a temperature above 50 °C and filtering the resulting mass. Basic salts are made similarly with the exception that a stoichiometric excess of the metal is used.

Preferably, overbased magnesium salicylate is used. These compositions can be characterized by their total base number (TBN). The total base number is preferably at least 100 mg KOH/g, more preferably at least 200 mg KOH/g, most preferably at least 300 mg KOH/g. The total base number is preferably at most 600 mg KOH/g. Another

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method of characterising overbased magnesium salicylates is by the magnesium content relative to the stoichiometrically equivalent amount of magnesium based on the amount of total acid present. Overbased magnesium salicyclates for use in the present invention preferably have a magnesium content of more than 500% of the stoichiometrically equivalent amount of magnesium based on the amount of total acid present, more preferably at least 550%.

The salicylate can be either substituted or unsubstituted. Suitable substituents include aliphatic groups containing from 1 to 40 carbon atoms and optionally containing one or more oxygen and/or nitrogen atoms, and hydroxy groups. Preferred substituents are alkyl groups containing from 6 to 30 carbon atoms, preferably from 12 to 20 carbon atoms. Preferably, the substituents are linear. The salicylate can contain from 1 to 4 substituents, preferably from 1 to 3, most preferably 1 or 2 substituents. Most preferably, the salicylate is substituted by 1 linear alkyl group containing from 14 to 18 carbon atoms.

Generally, mono-alkyl salycylic acids are prepared by alkylation of phenol and subsequent carboxylation.

Therefore, a small amount (generally at most 20 %mol) of dialkyl salicylate and unsubstituted salicylate can be present in the mono-alkyl salicylate.

Magnesium salicylate which can be used in the present invention, is commercially available. A suitable commercial product is SAP 007 (ex Infineum). The magnesium salicylate will usually be supplied in combination with mineral oil. The amounts referred to in

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this document, relate to the compound per se without mineral oil.

A process by which suitable magnesium salicylate can be prepared, has been described in US-A-4,627,928.

Zinc dithiophosphate is well known in the art.

Preferred dithiophosphate for use in the present
invention are represented by the following formula

are represented by the following for

$$O-R^9$$
 $R^8 - O - P = S$

(formula II)

 S^7

in which R⁸ and R⁹ can be either hydrogen or an aliphatic group. The aliphatic group is preferably a hydrocarbyl group. The hydrocarbyl group optionally contains an acid, a hydroxy and/or an ester group. The hydrocarbyl group is preferably an alkyl containing up to 12 carbon atoms optionally further containing an acid, a hydroxy and/or an ester group. The hydrocarbyl substituted dithiophosphate can contain 1 or 2 aliphatic groups, or it can be a mixture of dithiophosphates having 1 aliphatic group and dithiophosphates having 2 aliphatic groups. Preferably, the zinc dithiophosphate is a zinc dialkyl dithiophosphate.

Substituted dithiophosphates can contain primary, secondary and/or tertiary aliphatic substituents, i.e. substituents which are attached to the oxygen atom via a carbon atom attached to one, two or three further carbon atoms, respectively. In the present invention, it is preferred that at least 60 %wt of the aliphatic, preferably alkyl, substituents of the zinc dithiophosphate, are primary aliphatic substituents. More

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preferably at least 80 %wt of the aliphatic substituents are primary aliphatic substituents.

Suitable zinc dithiophosphates which are commercially available include Elco 108 (ex Elco Corporation), Lz 1375 (ex Lubrizol), OLOA 4269Q (ex Oronite) and HITEC 680 (ex Ethyl).

The amount of magnesium salicylate which is present is preferably at most 1 %wt, based on total composition, more preferably at most 0.5 %wt, most preferably at most 0.3 %wt. The amount of magnesium salicylate which is present is preferably at least 0.001 %wt, more preferably at least 0.005 %wt more preferably at least 0.01 %wt, most preferably at least 0.015 %wt. The amount of zinc dithiophosphate which is present is preferably at most 5 %wt, based on total composition, more preferably at most 3 %wt, most preferably at most 1 %wt. The amount of zinc dithiophosphate which is present is preferably at least 0.05 %wt, more preferably at least 0.7 %wt, most preferably at least 0.7 %wt, most preferably at least 0.7 %wt, most

The hydraulic fluid can further comprise (c) from 0.001 to 5 %wt of a rust inhibitor based on total composition, preferably a carboxylic acid or ester or amide or imide containing compound, more preferably a dicarboxylic acid or its mono- or di-ester or its mono-amide or di-amide or imide, more preferably a dicarboxylic acid or its ester, amide or imide containing a total number of between 4 and 70 carbon atoms. More preferably, the number of carbon atoms of the compound is between 20 and 40. A preferred compound is an aliphatic succinimide or succinic diamide. Such compounds have been described in EP-A-776964.

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Most preferably, (c) is from 0.001 to 5 %wt of a compound according to the following formula I

in which R_1 and R_2 are each hydrogen or alkyl or hydroxyalkyl of 1 to 30 carbon atoms; $R_{\scriptscriptstyle 3}$, $R_{\scriptscriptstyle 4}$ and $R_{\scriptscriptstyle 5}$ are each hydrogen or alkyl or hydroxyalkyl of 1 to 4 carbon atoms; X is CH or N and R_6 and R_7 are each hydrogen, alkyl or alkenyl of 1 to 30 carbon atoms, or an acyl group derived from a saturated or unsaturated carboxylic acid of up to 30 carbon atoms. Preferably, R_1 and R_2 are each an alkyl of from 3 to 6 carbon atoms, $R_{\scriptscriptstyle 3}$, $R_{\scriptscriptstyle 4}$ and $R_{\scriptscriptstyle 5}$ are each hydrogen, X is N and $R_{\rm 6}$ and $R_{\rm 7}$ are each an alkyl of 15 to 20 carbon atoms or an acyl group derived from a saturated or unsaturated dicarboxylic acid containing 4 to 10 carbon atoms, at least one of $R_{\scriptscriptstyle 6}$ and $R_{\scriptscriptstyle 7}$ being an acyl group. Especially preferred is aspartic acid, N-(3carboxy-1-oxo-2-propenyl)-octadecyl-bis(2methylpropyl)ester. Such aspartic acid esters are commercially available. Processes for preparing such

The weight ratio in which the compounds of the present invention are present, can differ within wide ranges. Preferably, the hydraulic fluid comprises magnesium salicylate, zinc dithiophosphate and optionally a rust inhibitor in such quantities that the weight ratio of magnesium salicylate to zinc dithiophosphate is from 1:5 to 1:100 and the weight ratio of magnesium salicylate to rust inhibitor is from 1:0 to 1:50.

compounds having been described in EP-A-434 464.

The lubricant base oils present in the hydraulic fluid of the present invention, can be any base fluid

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which is suitable for use in hydraulic fluids. The base oil can be a natural or a synthetic lubricant base oil, or a mixture thereof. The natural oil can be an animal oil or vegetable oil, such as lard oil or castor oil, or a mineral oil such as liquid petroleum oils and solvent treated or acid treated mineral lubricating oil of the paraffinic, naphthenic, or mixed paraffinic/naphthenic type which may be further refined by hydrocracking and hydrofinishing processes and/or dewaxing. Synthetic lubricating oils include hydrocarbon oils and halosubstituted hydrocarbon oils such as polymerised and interpolymerised olefins. A suitable base oil contains poly-alpha-olefins, such as polydecene. Preferably, the base oil is a hydrocarbon base oil. More preferably, the base fluid is a mineral oil which contains less than 10 % by weight of aromatic compounds, preferably less than 5 % by weight, most preferably less than 3.0 % by weight, measured according to DIN 51378. It is further preferred that the base oil contains less than 1.0 %wt of sulphur, calculated as elemental sulphur, preferably less than 0.1 %wt, more preferably less than 0.05 %wt, measured according to ASTM D 4045. Such mineral oils can be prepared by severe hydroprocessing. Preferably, the lubricating oil has a kinematic viscosity in the range of from 5 to 220 cSt at 40 °C, more preferably of from 10 to 200 cSt, most preferably of from 20 to 100 cSt.

The hydraulic fluid according to the present invention can contain further additives usually present in hydraulic fluids, such as pour point depressants, anti-foam agents and demulsifier. Pour point depressants generally are high molecular weight polymers such as alkylaromatic polymers and polymethacrylates. As anti-

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foam agents, silicone polymers and/or polymethacrylates are generally used. Demulsifiers which are generally applied are polyalkylene glycol ethers. Furthermore, further detergents such as sulphonates and phenates, metal deactivators, antioxidants such as phenolic compounds, diphenyl amines and phenyl naphthyl amines, ashless anti-wear agents and/or ashless dispersants, such as succinimides, can be present.

The hydraulic fluids of the present invention contain magnesium salicylate. For specific applications, it can be advantageous that the fluids contain a combination of magnesium and calcium salicylate.

The components of the present invention can be added per se to a lubricating oil, or they can be mixed into an additive package before being added to the lubricating oil. The additive package for preparing the hydraulic fluid preferably contains magnesium salicylate, zinc dithiophosphate and optionally a dicarboxylic acid or its mono- or di-ester or its mono-amide or di-amide or imide containing in total between 4 and 70 carbon atoms, wherein the weight ratio of magnesium salicylate to zinc dithiophosphate is from 1:5 to 1:100 and the weight ratio of magnesium salicylate to dicarboxylic acid or its mono-or di-ester or its mono-amide or di-amide or imide is from 1:0 to 1:50.

EXAMPLES

Formulations were prepared containing the following additives:

Magnesium salicylate: highly overbased magnesium alkylsalicylate having a total base number of 337 mg KOH/g (SAP 007 ex INFINEUM) and a magnesium content of about 750% of the stoichiometrically equivalent amount of

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magnesium based on the amount of total acid, containing 40 %wt of mineral oil

Calcium salicylate: overbased calcium alkylsalicylate having a total base number of 168 mg KOH/g (SAP 001 ex INFINEUM), containing 40 %wt of mineral oil.

The amounts of metal salicylate mentioned in Table 1, are the amounts of metal salicylate per se, without mineral oil.

Zinc dithiophosphate: zinc di(ethyl-

hexyl)dithiophosphate (Elco 108 ex Elco corporation)
Compound according to formula I: aspartic acid, N-(3-carboxy-1-oxo-2-propenyl)-N-octadecyl, bis(2-methylpropyl)ester.

These compounds were added to a lubricant base oil containing less than 1.0 % by weight of aromatic compounds, less than 0.05 %wt of sulphur, calculated as elemental sulphur and having a kinematic viscosity in the range of 32 cSt (at 40 °) (ISO viscosity grade 32).

The compositions obtained are described in Table 1. The amounts are of the compounds per se, excluding the mineral oil.

Compositions 1 and 2 were used in Vickers V104C vane pump tests. A new pump cartridge was used for each test. The test duration was 250 hours, performed at a fluid temperature of about 66 °C, at a fluid outlet pressure of 35 bar (3.5 MPa) and at a pump speed of 1450 revolutions per minute. The results are described in Table 1.

Table 1

		Composition 1	Composition 2
	magnesium salicylate	0.06 %wt	-
	calcium salicylate	-	0.06 %wt
5	zinc dithiophosphate	0.37 %wt	0.37 %wt
	compound of formula I	0.10 %wt	0.10 %wt
	lubricant base oil	balance	balance
	Low load test		
	ring weight loss (mg)	13	96
10	vane weight loss (mg)	2	1
	total weight loss (mg)	15	97
	From Table 1 it is clear	that better prot	ection against
	wear is provided by a co	mposition contain	ing magnesium
	salicylate (1), than by	a composition con	taining calcium
15	salicylate (2). The ring	g weight loss and	l total weight
	loss incurred when using	composition 1 is	significantly

less than that incurred when composition 2 is employed.

The vane weight loss is similar for both compositions.

TS7564.DOC

I CLAIM:

- 1. A hydraulic fluid comprising a lubricant base oil in combination with
- (a) from 0.001 to 5 %wt of magnesium salicylate,
- (b) from 0.01 to 8 %wt of zinc dithiophosphate.
- 2. A hydraulic fluid according to claim 1, further comprising (c) from 0.001 to 5 %wt of a dicarboxylic acid or its mono- or di-ester or its mono-amide or diamide or imide containing in total between 4 and 70 carbon atoms.
- 3. A hydraulic fluid according to claim 2, in which (c) is a compound according to the following formula I

in which R_1 and R_2 are each hydrogen or alkyl or hydroxyalkyl of 1 to 30 carbon atoms; R_3 , R_4 and R_5 are each hydrogen or alkyl or hydroxyalkyl of 1 to 4 carbon atoms; X is CH or N and R_6 and R_7 are each hydrogen, alkyl or alkenyl of 1 to 30 carbon atoms, or an acyl group derived from a saturated or unsaturated carboxylic acid of up to 30 carbon atoms.

4. A hydraulic fluid according to claim 1, comprising magnesium salicylate, zinc dithiophosphate and optionally a compound according to formula I in such quantities that the weight ratio of magnesium salicylate to zinc dithiophosphate is from 1:5 to 1:100 and the weight ratio of magnesium salicylate to rust inhibitor is from 1:0 to 1:50.

- 5. A hydraulic fluid according to claim 1, which fluid further comprises pour point depressant, anti-foam agent and/or demulsifier.
- 6. Additive package for preparing a hydraulic fluid which additive package comprises magnesium salicylate, zinc dithiophosphate and optionally a dicarboxylic acid or its mono- or di-ester or its mono-amide or di-amide or imide containing in total between 4 and 70 carbon, wherein the weight ratio of magnesium salicylate to zinc dithiophosphate is from 1:5 to 1:100 and the weight ratio of magnesium salicylate to dicarboxylic acid or its mono-or di-ester or its mono-amide or di-amide or imide is from 1:0 to 1:50.

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HYDRAULIC FLUID

Abstract of the Invention

A hydraulic fluid comprising a lubricant base oil in combination with (a) from 0.001 to 5 %wt of magnesium salicylate, (b) from 0.01 to 8 %wt of zinc dithiophosphate, and optionally (c) from 0.001 to 5 %wt of a dicarboxylic acid or its mono- or di-ester or its mono-amide or di-amide or imide containing in total between 4 and 70 carbon atoms; and an additive package comprising magnesium salicylate, zinc dithiophosphate and optionally a dicarboxylic acid or its mono- or di-ester or its mono-amide or di-amide or imide containing in total between 4 and 70 carbon.

DECLARATION AND POWER OF ATTORNEY FOR PATENT APPLICATION

ATTORNEY'S DOCKET NO. TS6574 (US)

As a below named inventor, I hereby declare that:

inventor matter v	r (if only one name is l	ess, and citizenship are as stated bel isted below) or an original, first and r which a patent is sought on the inv lowing box is checked:	l joint inventor (if plural name	s are listed below) of the subject
	was filed onNumber	as United States Application and was amended on	Number or PCT International A	Application
	y state that I have reved by any amendment re	iewed and understand the contents ferred to above.	of the above-identified specia	fication, including the claims, as
I ackno	wledge the duty to discl	ose information which is material to	patentability as defined in 27 C	CFR § 1.56.
invento States,	r's certificate, or § 3650 listed below and have a	y benefits under 35 U.S.C. § 119((a) of any PCT International applications identified below by checking the n having a filing date before that of the state of t	tion which designated at least of box, any foreign application f	one country other than the United for patent or inventor's certificate, y is claimed. Priority
	FOREIGN APPLICATI		D 11/2 (0) 12/13/15 (D	Not Claimed
APPLI	CATION NUMBER	COUNTRY	DAY/MONTH/YEAR	FILED
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(NAME) <u>Dennis V. Carmen</u> c/o Shell Oil Company Intellectual Property P.O. Box 2463 Houston, TX 77252-2463



DECLARATION AND POWER OF ATTORNEY FOR PATENT APPLICATION

ATTORNEY'S DOCKET NO. TS7564 (US)

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

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FOURTH INVENTOR'S SIGNATURE	DATE SIGNED
POORTH INVENTOR 5 SIGNATURE	
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FIFTH BUENTOD'S SIGNATURE	DATE SIGNED
FIFTH INVENTOR'S SIGNATURE	DATE SIONED
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